Pub. 10.11.82

(12) UK Patent Application (19) GB (11) 2 097 672 A

(21) Application No 8113585
(22) Date of filing 1 May 1981
(43) Application published 10 Nov 1982
(51) INT CL[3] C07J 71/00 A61K 35/78
(52) Domestic classification A5B N C2U 4A1B 4D2 4N12
(56) Documents cited GB 1239555 GB 0974876 GB 0799047 GB 0797384 GB 0733052 GB 0728961 GB 0722186 GB 0702072 GB 0537794
(58) Field of search A5B C2U

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(54) Process for extraction of crude sapogenins from Agave Leaves

(57) A method for the preparation of a sapgoenin-containing material, which method comprises hydrolysing juice obtained from the leaves of the Agave plant by heating the juice in acid solution, the acid concentration and period of hydrolysis being sufficient to hydrolyse approximately half of the sugar moieties present, and then adding to the product a further quantity of acid and heating the mixture to complete the hydrolysis.

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SPECIFICATION

Process for extraction of crude sapogenins from agave leaves

The present invention relates to the extraction of sapogenins from plant juices and specifically to the extraction of the sterioidal sapogenins hecoqenin and tiqoqenin from the leaves of sisal henequen and the like (*Agave Sisalana* and related species).

There are several problems associated with the extraction of sapogenins from Agave Leaves. This is because it is necessary for the process to provide for sapogenin recovery and fiber recovery for maximum recovery of both these products, in a way which is financially feasible. During the conventional process for decortication of agave leaves to separate the fibre from the pulp, large quantities of water are used to maintain high fibre yields. This results in the dilution of the juice obtained from the pulp, to such an extent that it is not a practical financial proposition.

In one aspect of this invention sapogenin containing material is extracted from the leaves of the Agave plant by treating the leaves to separate the plant juice and the leaf fibre, in the substantial absence of added water, and hydrolysing the juice obtained to produce a sapogenin-containing material. This aspect of the invention involves dry decortication of the Agave Leaves. During this operation there is undiluted juice available, but the juice does contain a small amount of fibre. As will be discussed later, this loss can be turned into an advantage.

The invention also provides a method for the preparation of a sapogenin-containing material, which method comprises hydrolysing juice obtained from the leaves of the Agave plant by heating the juice in acid solution, the acid concentration and period of hydrolysis being sufficient to hydrolyse approximately half of the sugar moieties present, and then adding to the product a further quantity of acid and heating the mixture to complete the hydrolysis.

In the preferred embodiment, the proposed process for the recovery of sapogenins may be divided into five parts a). Juice extraction from the pulp, b.) two stage hydrolysis, c). Filtering of hydrolysate, d) Drying of cured hydrolysate sludge), e) Grinding and packing of the finished material, each of which is explained in more detail below.

(a) *Juice Extraction:*

As mentioned above, the dry decortication method is preferable because it results in smaller quantities of undiluted juice. Normal decortication methods produce a 4 to 5 fold juice dilution, which leads to a corresponding increase in capital cost of equipment, heating costs, and the cost of the acid used, as compared with the preferred method of the invention. Conventional decorticating machinery may be used except that no water is supplied to the machines. The leaves thus produce two products, sisal fibres, and leaf pulp, from which juice may be obtained by squeezing.

However, dry decortication of the Agave Leaves result in a slight loss of fibre, this fiber being mixed with the pulp. Once the pulp has been utilized for obtaining the juice, the residual material can be processed to obtain short fibres of between 2 inches and 7 inches. These can be dried in the sun and used as a stuffing material for Cushions, Mattresses etc., enabling the Sisal Plantation to recover the loss resulting from the diy decortication method.

It has been found that to obtain a final product which is high in hecoqenin and low in tiqoqenin, the pulp should preferrably come from the portion of the leaf between 11 inches and 17 inches from the butt ends of the leafs, and from leaves which are over two years old. Using such materials we have been able to obtain juice with tiqoqenin levels of below 6% (of the sapogenins). The pulp thus obtained is passed through a roller press which results in the removal of as much as 80% of juice content of the pulp. If necessary the pulp can be passed through two consecutive rollers to maximize the extraction of the juice. The obtained juice is then collected over a simple fine mesh filter to remove as much coarse debris as possible. When enough juice is collected in the storage of holding tank, it may be transferred to the next process point i.e. a hydrolysis tank.

b) *The Two-stage Hydrolysis Process*

The hydrolysis process involves boiling of the juice obtained with a suitable quantity of an acid. The steroidal saponin present in the juice has two or more sugar moieties attached to it, and this makes it soluble in water. In order to obtain the sapogenin from the saponin the whole of the sugar portion has to be hydrolyzed. However if the hydrolysis is carried out by adding all the acid required for hydrolysis at the beginning of the reaction, strong acidic conditions (2N or stronger) and prolonged boiling of the acidified juice (between 20 and 30 hours) are required. Prolonged boiling causes large quantities of tars, resins and other undesirable plant materials to be precipitated with sapogenin from the highly acidified juice, and these side reactions in turn consume valuable acid. Prolonged heating also results in high energy costs greater 'wear and tear' on the process equipment due to highly acidic conditions, as well as lowered product quantity and quality because of the larger amounts of undesirable plant materials recipitated.

The two-stage hydrolysis reaction avoids these difficulties by lowering the maximum acid concentration experienced by the solution.

The acid used may referably be sulphuric or hydrochloric, the preferred quantity of sulphuric being 1.5% (v/v) and that of hyrochloric being 3.0% (v/v).

In a preferred embodiment, the hydrolysis method of the invention is carried out as follows. When the juice is ready at the storage tank, it is pumped into a first stage hydrolysis vessel. This vessel is preferably made of acid resistant fibre-glass, with a capacity of about 6000 litres. The heating system in the first stage hydrolysis vessel preferably consists of a heating coil for example of copper connected to a steam-line. It has been found that a coil of 2½ feet

diameter and 3½ feet in height is sufficient. The coil is located inside the bottom section of the vessel which is conical. Once the vessel has juice reaching over the level of the coil, the steam line is turned on and the juice starts to heat up. As soon as the vessel is filled to capacity the acid can be added. Thus in a vessel containing 6000 litres of juice, about 180 litres of hydrochloric acid (30%) is added, or 90 litres of sulphuric acid (15%). After the juices has reached boiling point, it is allowed to boil for between 8 hours and 14 hours. This results in the completion of the first stage of hydrolysis, after which approximately half the sugar moieties attached to the sapogenin have been removed by hydrolysis, and a heavy precipitate of partially hydrolysed saponin is formed.

The steam line is turned off and the surface of the juice (or the vessel top itself) is covered with a suitable lid. This results in the settling down of the precipitated material to the bottom part of the tank, the settling process requiring about 4 hours to 6 hours. At the same time the partially hydrolysed juice cools down a bit. When the settling is complete the supernatant liquid is removed and discarded. (In practice, the level formed by the semi-solid is readily reproducable and a tap can be inserted just above this level). It has been found that 15% to 20% of the initial volume of juice remains as the semi-solid volume. Several points are relevant as regards to the first stage: It is important to keep the juice boiling constantly so that the cycle is as short as possible. The lid to be used for the settling process is preferably an insulating one to stop large heat losses from the surface which would otherwise cause convection current to circulate the solid resulting in excessive times for settling (even up to 3 days).

The semi-solid may then be pumped into another vessel in preparation for the second stage of the hydrolysis process. The semi-solids concentrates from 5-6 batches may be combined into a single batch to give about 6000 litres.

The second stage of the hydrolysis process may be carried out in a way generally similar to the first stage. Moreover because of the solids produced in the second stage of hydrolysis, heating in the second stage hydrolysis is preferably carried out using an open ended pipe for live steam heating. This has been found to be much more efficient in practice than a coil, since with the latter, the solids cover the coil and encrust it after one or two batches resulting in much reduced heat efficiency. As soon as the second stage vessel is filled to capacity the steam is turned on and acid is added in the same quantities as the first stage hydrolysis process, i.e. if sulphuric acid then 1.5% and if hydrochloric 3.0%.

After the concentrate has reached boiling point it is boiled for from 8 hours to 12 hours, when the hydrolysis is complete. The thick hydrolysate is allowed to cool without covering the vessel, to about 50 to 60°C when it can be transferred to a storage vessel ready for the filtering stage.

c) *Filtering of Hydrolysate*

It is possible to use complex filtering systems at this stage of the process, such as a filter press. We have found in practice that a cheap and simple device such as a polypropylene filter bag is quite efficient and almost maintenance free, it has been found that about forty bags of 4 feet by 3 feet each are enough for one batch of second stage hydroysate. The bags are simply hung on a filtering line, filled with hydrolysate and left to carry out the filtering. Normally in about 36 hours the filtering is complete and the bags are found to contain a thick sludge which is ready for the next stage of drying

d) *Drying of Crude Hydrolysate Sludge*

There are two preferred methods of drying: a) open sum drying. b) tray drying. The first method depends of course on the availability of sunshine whilst the second method can be used in any weather. In this latter method floor trays of cement for example 8 feet by 4 feet and 6 inches deep may be constructed containing a heating element consisting of longitudinal steam pipes e.g. of copper. The thick sludge is simply poured onto the element in the tray and the steam turned on. The drying process is completed within another 36 hours to 48 hours depending of course on the water content of the sludge. The first method, i.e. sun drying, may be used in conjunction with the second method that is, the sludge is only partially dried in the trays and then removed to open air trays where it slowly dries. It has been found that in practice this combined method results in a good product.

e) Grinding and packaging: At this stage the crude hydrolysate is solid and in lumps with a "Coffee Grounds" colour. It smells faintly of acid and is slightly corrosive. This is due to the fact that there is no neutralizing step in the process, and a neutral product is probably not required at this stage. The last step is to convert the lumps into fine powder to facilitate processing and also packaging. The powder can simply be packed in a polythene bag with a hessian bag around it.

In the preferred practice of the invention the pulp from dry decortication is taken only from 11 inches of the butt ends of the leaves and then only from leaves which are over 3 years old (i.e. 3rd cut and over). The pulp is squeezed through corrugated roller presses, and the juice filtered and pumped into fibreglass vessels with a capaicty of 6,000 litres and containing a heating coil of copper. The juice is boiled with 3% 9v/v hydrochloric acid (33%) for 12 hours allowed to settle for 6 hours. The supernatant liquid is drained and "the bottoms" are combined with another four first stage bottoms, in a second stage hydrolysis vessel which has an open copper steam pipe for 'live steam' heating. This second boiling is carried out with more 3% (v/v) hydrochloric acid (33%) for another twelve hours. The thick hydrolysate is allowed to cool for about 6 hours and then put into polypropylene filter bags. The filtering being complete in 36 hours, the sludge is removed to drying trays where it is dried for about 36 hours. The soft solids are then removed to outside open air trays and allowed to dry further reducing the water contents to less than 10% (w/w). The solid hydrolysate is then ground to a powder and packed.

CLAIMS

1. A method for the preparation of a sapogenin-containing material, which method comprises hydrolysing juice obtained from the leaves of the Agave Plant by heating the juice in acid solution, the acid concentration and period of hydrolysis being sufficient to hydrolyse approximately half of the sugar moieties present, and then adding to the product a further quantity of acid and heating the mixture to complete the hydrolysis.

2. A process as claimed in claim 1, wherein the juice is obtained by decortication of the Agave leaves, in the substantial absence of added water.

3. A process as claimed in claim 1 or claim 2, wherein the juice is obtained from the butt ends of Agave Leaves, the length of these butt ends being from 11 to 17 inches.

4. A process as claimed in any one of the preceding claims wherein the pulp obtained is from Agave Leaves which are at least 3 years old.

5. A process as claimed in any one of the proceding claims wherein the partially hydrolysed material is allowed to settle and the supernatant liquid is removed, before addition of the second quantity of acid.

6. A process as claimed in any one of the preceding claims wherein the acid used for the first stage hydrolysis is approximately 30% hydrochloric acid at 3% (volume of acid/volume juice) or approximately 30% sulphuric acid at 1.5% (volume acid/volume juice).

7. A process as claimed in any one of the preceding claims wherein the acid used for the second stage hydrolysis is approximately 30% hydrochloric acid at 3% (volume of acid/volume juice) or approximately 30% sulphuric acid at 1.5% (volume acid/volume juice).

8. A process for producing sapogenin-containing material substantially as herein before described.

Printed for Her Majesty's Stationery Office, by Croydon Printing Company Limited, Croydon, Surrey, 1982.
Published by The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from which copies may be obtained.